Title	Determination Carboxylic Groups in Vinylon Fiber
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drawing.

Some determination of contraction of filament through wet and dry heat have also been carried out. By wet heat at 90°C filament drawn to nine times of its original length contracts almost completely to its original length. By dry heat at 250°C filament drawn as high as five times, contracts competely.

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37. On the Heat Decomposition of of Polyvinyl Alchol Fiber

Kiyoshi Hirabayashi and *Jun Hiramatsu*

(Sakurada Laboratory)

X-ray investigation of the process of heat decomposition of polyvinyl alcohol (P. V. A.) fiber have been carried out. After the heat treatment (200°C, 7 min.) P. V. A. were dried (110°C, 24 hrs.) and subjected to heat decomposition in an electric oven. The oven was heated from 150°C, and 415°C, and during the heating fibers were not allowed to contract. The process may be divide into four stages. 1), growth of crystallites between 150° and 200°C. 2), growth of crystallites and their disorientation between 200° and 250°C. 3), melting of crystallites and decomposition between 250° and 350°C. 4), carbonization above 350°C. Rate of the weight loss of the sample due to dehydration was the greatest between 250° and 300°C.

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38. Determination Carboxylic Groups in Vinylon Fiber

Kiyoshi Hirabayashi and *Shoji Yamamoto*

(Sakurada Laboratory)

Determination of carboxylic groups of Vinylon fiber have been carried out according to the method of reversible methylene-blue absorption. Polyvinyl alcohol (P. V. A.) fiber is subjected to heat-treatment (5 min. at ca. 215°C), then partially (35-40 mol %.) formalized so Vinylon is obtained. As is shown in table 1, various preteatment gives different results, so we cannot determine the true contents of the carboxylic groups of the Vinylon fibsr. But it seems, that this method is useful for the technical characterization of Vinylon fiber.

The table 2 shows M. B. absorption of Vinylon after bleaching.

Table 1. Determination of carboxylic groups under various condition.

No.	Substance and pretreatment.	mg. M.B./Subst.	Mol. vinylgroup. /mol. COOH
1.	P. V. A. fiber.	3.02	2420
2.	No. 1. after the heat-treatment.	1.099	6600
3.	No 2. after formalization (Vinylon)	0.250	27500
4.	After immersion of No. 2 in water (40°C, 24 hrs.)	1.129	5650
5.	After immersion of No. 3 in water (45°C. 17 hrs.)	0.472	14600
6.	After immersion of No. 3 in boiling water (1 min.)	0.624	11200
7.	〃 (5 min.)	0.695	10000
8.	〃 (10 min.)	0.777	8990
9.	〃 (30 min.)	1.028	6690
10.	〃 (60 min.)	2.38	2940
11.	〃 (120 min,)	2.30	3010

Table 2. Determination of carboxvlic groups after bleaching.
(The original sample is No. 3 of table 1.)

Bleaching agent.	Condition of bleaching	mg. M.B./g. Subt.	Mol. vinylgroup. /Mol. COOH
Bleaching-powder	Cl_2 1g/L,N/1oH_2SO_4, 10c, 17 hrs.	0.851	8100
	Cl_2 1g/L, CH_3COOH, 1g./L 17 hrs.	1.73	3960
	Cl_2, 3.4g/L, CH_3COOH, 1g./L, 17 hrs.	0.803	8580
	Cl_2, 5g/L, CH_3COOH, 1g./L, 17 hrs.	9.12	760
H_2O_2	g./L, 45°C, 17 hrs, PH=8.0	0.460	15000
〃	50g./L, 45°C 17 hrs, PH=8.0	1.345	5090
K-permangan.	2g./L, 10°C, afterwards immersed in oxalic acid, washed with water, dried.	0.510	13680
Sodiumchlorite.	5g./L, 45°C, 17 hrs, without CH_3OOH.	0.530	13040
	1g./L, 45°C, 17 hrs, CHCOOH 1g./L.	0.952	7240
	5g·/L, 10°C, 17 hrs, without CH_3COOH	0.576	11960
	5g./L, 10°C, 17 hrs, CH_3COOH 1g./L.	0.438	15620
Sodium hydrosulphite.	5g./L, 45°C, 17 hrs,	0.741	9300
	50g./L, 45°C, 17 hrs,	0.973	7070

39. On the Low Formalization of Polyvinyl Alcohol Fiber

Ichiro Sakurada and *Naofumi Nakamura*

(Sakurada Laboratory)

By the manufacture of synthetic fiber Vinylon, polyvinyl alcohol fiber, which has been subjected to heat treatment, is formalized ordinary with a bath of the following composition: H_2SO_4 250g/L, Na_2SO_4 300g/L, HCHO 60g/L.

For the purpose of utilization of the wash liquor, formalization of the fiber with this wash liquor havs been undertaken. The liquor have the following composition: H_2SO_4 80~150 g/L, Na_2SO_4 100g/L, HCHO 10-2 g/L. It have been found that polyvinyl alcohol fibers can be easily formalized with this dilute bath and formaldehyde is almost exhausted. This process may have a practical application.